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Research Article

Structural and Optical Characterisation of Zinc Doped Potassium Hydrogen Tartrate Crystal for Antibacterial Analysis

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Abstract: Several researchers have grown materials in single crystal form by gel technique and doped these materials by suitable dopant to determine the effect of doping on their characteristics. Among the several tartrate compounds, ammonium hydrogen tartrate and rubidium hydrogen tartrate have received greater attention because of their applications in transducers and in several linear and non-linear devices. In the present study, Growth of Zinc doped potassium hydrogen tartrate single crystal by gel technique is reported. The crystal is characterized by using EDAX, PL,UV-vis, powder XRD, FTIR, RAMAN and ANTI BACTRIAL analysis.

Keywords: Gel growth, EDAX, PL, UV-vis, powder XRD, FTIR, RAMAN and ANTI BACTRIAL analysis.

INTRODUCTION

Several tartrate compounds are of considerable importance because of their interesting physical properties such as dielectric, piezoelectric, ferroelectric and optical second harmonic generation [1-4]. These characteristics of tartrate compounds are exploited for their use in transducers and in several linear and non-linear mechanical devices [1, 3]. They are also used in non-linear optical devices, optical transmission characteristics, fabrication of crystal oscillators and controlled laser emission [5-7]. As tartrate compounds are sparingly soluble in water and are thermally less stable, it is convenient to grow these materials by gel technique. [8-10].

Alkali metals, inspite of having important metallic properties such as high electric and thermal conductivity, have received less attention to the growth and characterization of these elements as metallic tartrate. Alkali metals are more metallic in character than alkaline earth and transition metals because the valence shell electron of the former is loosely bound to their atoms [11]. However, information about growth, characterization and properties of other transition metal tartrate such as Zn doped potassium hydrogen tartrate in the form of single crystals is not available in the literature.

Therefore, it was felt worthwhile to grow other transition metal tartrate in the form of single crystals and study their properties which may lead to an information about their application in different electronic devices. The present study deals with the growth of potassium hydrogen tartrate by gel technique and its characterization.

An attempt was, therefore, made to grow single crystals of modified composition by doping potassium hydrogen tartrate with other transition metals such as Zn. Response of a material on modification of its composition by doping foreign ions provides very useful information. It is observed that the doping of foreign ion into pure potassium hydrogen tartrate crystals affects different characteristics of the parent material. To the best of authors knowledge doping by foreign elements like Zn in potassium hydrogen tartrate grown by gel technique is reported for the first time.

EXPERIMENTAL

Growth Procedure

Gel technique was employed for the growth of doped potassium hydrogen tartrate crystal at room temperature. This technique consists of incorporating one reactant in the gelling mixture and then diffusing another reactant into the gel. This leads to high supersaturation, the initiation of nucleation and finally crystal growth.

The crystallization apparatus for the growth of doped potassium hydrogen tartrate consists of borosilicate glass tubes of length 15 cm and diameter 2.5 cm placed vertically on a stand. Silica gel (sodium meta silicate solution) is used as the growth medium. 10 ml of double distilled water is added with 1M of sodium meta silicate. 10 ml of tartaric acid 1M is added to form the gel medium. The test tubes were sealed with some suitable material to prevent fast evaporation and contamination of the exposed surface of the gel.

The gel setting time was found to be strongly dependent on pH and environmental temperature. It would take about 24 hours for the gel to set in summer (35-40ºC) whereas it would take even 14 days for the gel to set in winter (10-15ºC). After confirming the gel setting, an aqueous solution of potassium iodide of (0.5-1.5) molarity was poured over the gel carefully along the walls of a test tube so as to avoid any gel breakage. The diffusion of K+ ions through the narrow pores of the silica gel lead to reaction between these ions and $HC_4H_4O_6^-$ ions present in the gel as lower reactant.

The reaction is expected to take place leading to the formation of pure potassium hydrogen tartrate crystals. To grow zinc doped potassium hydrogen tartrate crystals, potassium iodide solution was first mixed with an aqueous solution of zinc acetate of (0.2-0.8) M.

The diffusion of Zn+ and K+ ions through the narrow pores of the silica gel lead to reaction between these ions and the HC4H4O6- ions present in the gel as lower reactant. The reaction leads to the formation of zinc doped potassium hydrogen tartrate crystals. The doped crystal is transparent single crystal with a good morphology. The shape of doped single crystal is found to be orthorhombic

Characterization Technique

The single crystal of zinc doped potassium hydrogen tartrate were characterized by using X-ray powder diffraction, FTIR spectroscopy, Raman spectroscopy, Photoluminescence, EDAX analysis and Antibacterial activity.

RESULTS AND DISCUSSION

Energy Dispersive X-ray Analysis

EDAX stands for Energy Dispersive Analysis by X-rays and it is a technique used for identifying the elemental composition of the specimen. EDAX is a technique, which makes it possible to identify elements and know their concentration by analyzing the energies of X-ray photons emitted as a result of bombardment by an electron beam [12].The atomic and weight % of Zinc doped crystal is present in Table 1. Atomic and weight percentage as obtained on EDAX analysis of zinc doped crystal of potassium hydrogen tartrate**.**

Table-1: Atomic and weight percentage as obtained on EDAX analysis of zinc doped crystal of potassium hydrogen tartrate.

ELEMENT	WEIGHT %	ATOMIC %
C	30.09	43.40
O	41.95	45.42
Si	00.67	00.41
K	19.84	08.79
Zn	07.45	01.98

The weight [%] and atomic [%] calculated from peaks height further confirms the expected proportion of Carbon, Oxygen, Zinc and Potassium crystals. It is observed that the dopant of Zinc has entered into the lattice site of pure potassium hydrogen tartrate crystal.

In the present study the crystal was analyzed by scanning electron microscope (Model: HITACHI S-3000 H). The recorded EDAX spectrum is shown in fig. 1

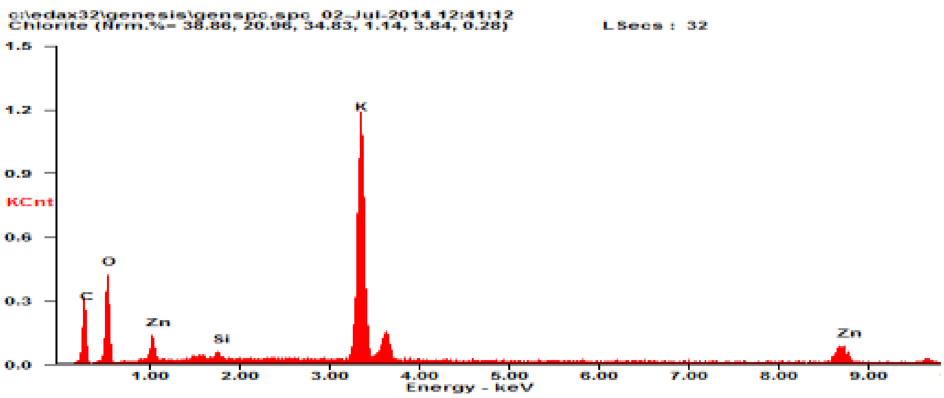

Fig. 1 EDAX analysis of Zinc doped Potassium Hydrogen Tartrate

From the spectrum it is confirmed that the elements such as C, O, K and Zn were present in the sample and it is noted here that the element H cannot be identified using the EDAX technique. Since the element H is a light element, it cannot be identified using EDAX method [13].

Pure potassium hydrogen tartrate crystals and Zn doped crystals were found to be more perfect in terms of transparency. This might be due to the slower reaction nature of K^+ and Zn^+ ions with $HC_4H_4O_6^-$ ions, respectively and hence the size of the crystal is small. The photographs of the Zinc doped crystals are shown in Figure.2

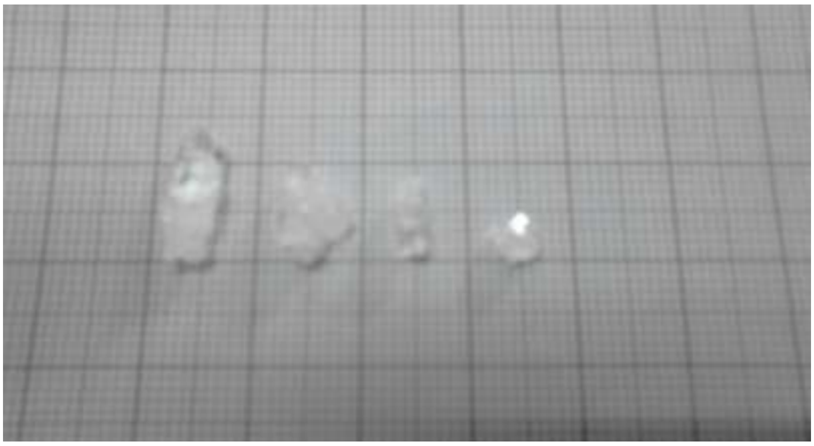

Fig-2: Crystal grown in gel Zn doped $KHC_4H_4O_6$

Photoluminescence

Photoluminescence spectra recorded between 320 nm and 520 nm for a zinc doped potassium hydrogen tartrate photoluminescence spectrum was performed using Varian Cary Eclipse Fluorescence spectrophotometer at Alagappa University, karaikudi. Spectrum shown in Fig. 3.

The room temperature Photoluminescence spectrum comprises outstanding violet emission peak at 390 nm, blue emission at 416 nm and one very scrawling blue shoulder peak at 476 nm. One green emission peak is identified at 508 nm [14].

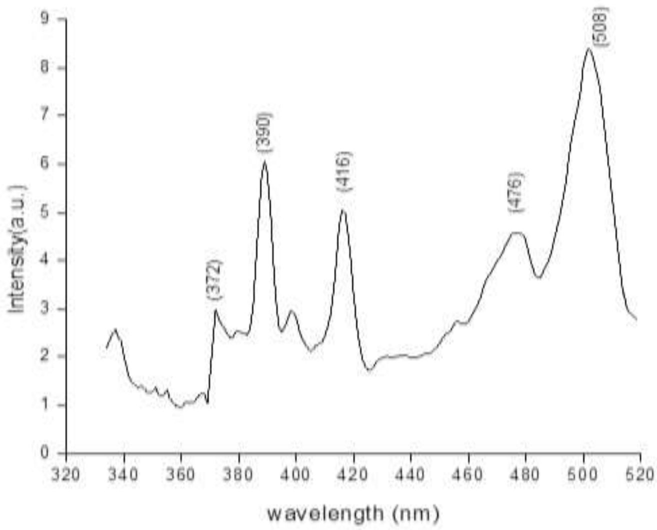

Fig-3: Photoluminescence spectrum of Zinc doped Potassium hydrogen tartrate crystal.

The emission spectrum shows the peaks at 390 nm, 416 nm, 476 nm, and 508 nm. Most intense peak is at 508 nm which is the green emission. Blue emission peak at 476 nm is less intense compare to the violet emission peak at 390 nm. Certain author assume that the green luminescence band with a maximum at λm = 535 nm.

UV-vis Spectroscopy

To find the transmission range of the grown crystal, the optical transparency of the grown crystals were analyzed by taking the UV-Visible spectra using Lambda-35 spectrometer between 200nm to 1100nm. A graph of percentage of transmission vs. wavelength is shown in fig. 4.

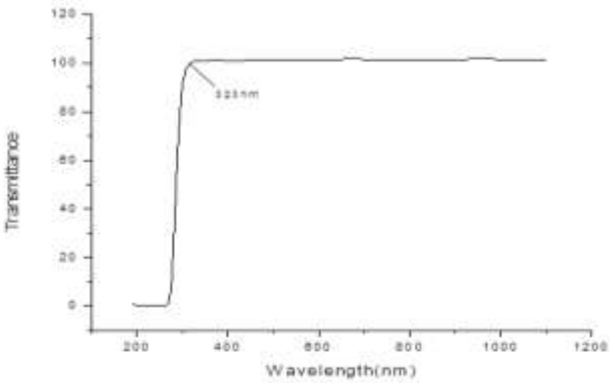

Fig-4: Transmittance graph

From the graph, the optical transparency of the grown crystals was about 100%.The grown crystals have an excellent transparency in the visible region, which is an essential parameter for optical applications. Absorption peak shown fig. 5.

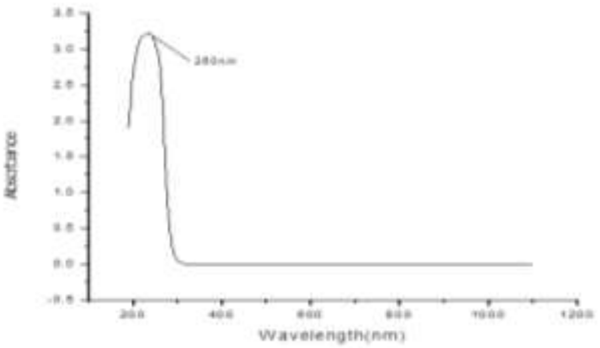

Fig-5: Absorbance graph

The absorption coefficient (α) were determined using Beer's law

$$\alpha = \frac{1}{d}log(\frac{1}{T})$$

An absorption coefficient (α) obeying the following relation for high photon energies.

$$\alpha = \frac{A(h\vartheta - E_g)^2}{h\vartheta}$$

Where E_g is optical band gap of the crystal and A is a constant. The plot of variation of Photon energy (E) in eV and $(\alpha h\nu)^2$ is shown in Fig. 6

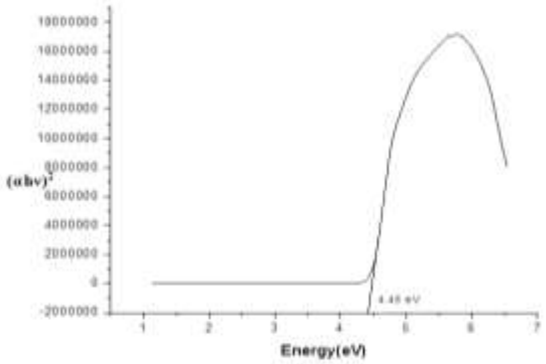

Fig. 6 Band gap curve of Zinc doped $KHC_4H_4O_6$

The linear region of the curve was extrapolated to find the x-intercept which gives the Band gap of the grown crystal. The band gap is found to be 4.45eV. This crystal can be a suitable material for the optoelectronic devices like LED and Laser diodes

FTIR and RAMAN

FTIR and Raman spectra recorded for zinc potassium hydrogen tartrate crystal. The presence of broad band in the wave number range 3200-3500 cm^{-1}. Indicates the presence of water of hydration in zinc potassium hydrogen tartrate crystal. Figure 7 and 8 shows the FTIR and Raman spectrum for Zn doped Potassium hydrogen tartrate crystal. In the case of Zinc doped potassium hydrogen tartrate ($KHC_4H_4O_6$). We have assigned the bands at 3315 cm^{-1}(IR)and 3288 cm^{-1}(Raman) to the OH stretching of H_2O.

A peak at 2974 cm^{-1}(IR) and 3044 cm^{-1} (Raman) has been assigned to the CH stretching in tartrate ion. The bands at 2894 cm^{-1} and 2988 cm^{-1} to the CH vibration of tartrate ion (TAYLOR et al.). The observed vibrational frequency and their assignment are presented in Table 2.

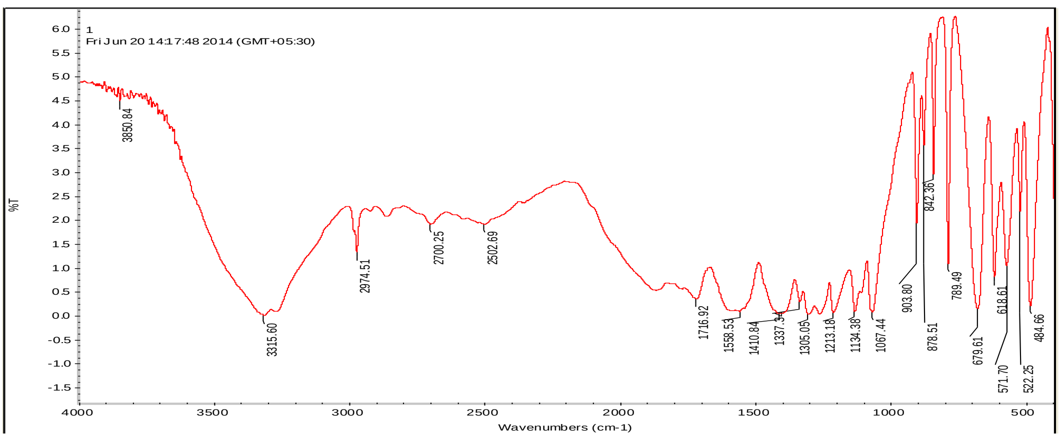

Fig-7: FTIR spectrum of Zinc Potassium hydrogen tartrate crystal.

The observed peaks at 1558 cm^{-1}(IR) and 1700 cm^{-1} (Raman) is due to the CO_2 asymmetric stretching and CO_2 stretching vibration.The IR bands observed at 1305 cm^{-1} have been assigned to the CH bend. The bands at 1213 $cm^{-1}$and 1134 cm^{-1} are due to the CO stretching mode.

The peaks at 1066 cm^{-1}(IR) and 1098 cm^{-1}(Raman)have been assigned to the CO stretching mode. 903 cm^{-1}(IR) are ascribed to the C-C stretching frequencies in the tartrate ion. The bands below 900 cm^{-1} (IR)and 781 cm^{-1}(Raman)are due to the CO_2 deformation and other internal modes of tartrate ions.

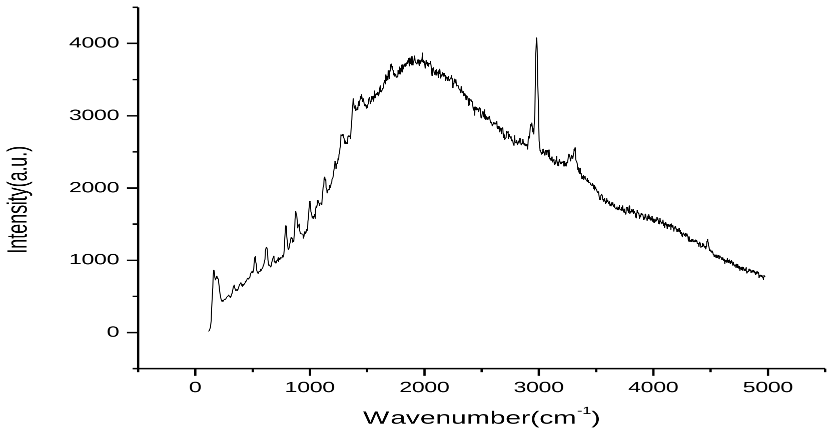

Fig. 8 Raman spectrum of Zinc Potassium hydrogen tartrate crystal

Table-2: **Comparative assignments of FTIR and Raman spectra of some prominent peaks corresponding to a particular functional group of pure and zinc potassium hydrogen tartrate crystals are given in table.**

Infrared	Raman	Assignments
3315	3288	OH stretching
2974	3044	CH stretch
1558	1700	C=O stretch
1410	1404	CO_2 bend
1337	-	CH bend
1305	-	CH bend
1213	-	CO stretching
1134	-	CO stretching
1067	1098	CO stretching
903	-	C-C stretching
842	-	CO_2 deformation
789	781	CO_2 deformation
522	-	CO_2 deformation
	452	CO_2 twist & C-C torsion
	112	CO_2 twist & C-C torsion

FTIR spectrum reveals the presence of O-H bonds, C-O bond, C-H bond, C-C bond and carbonyl C=O bonds. From the spectrum, it was found that although the radiations are absorbed at the same frequency by pure and doped crystals. The percentage of transmittance of Zn^{2+} doped $\mathbf{KHC_4H_4O_6}$ crystal is higher than the percentage of Pure $\mathbf{KHC_4H_4O_6}$ crystal compare the reference of pure crystal [15].

Powder X-ray diffraction:

The powder X-ray diffractograms of Zinc doped potassium hydrogen tartrate crystal is shown in Fig. 9. The crystallinity of Zinc doped crystal is quite clear from the diffractograms.

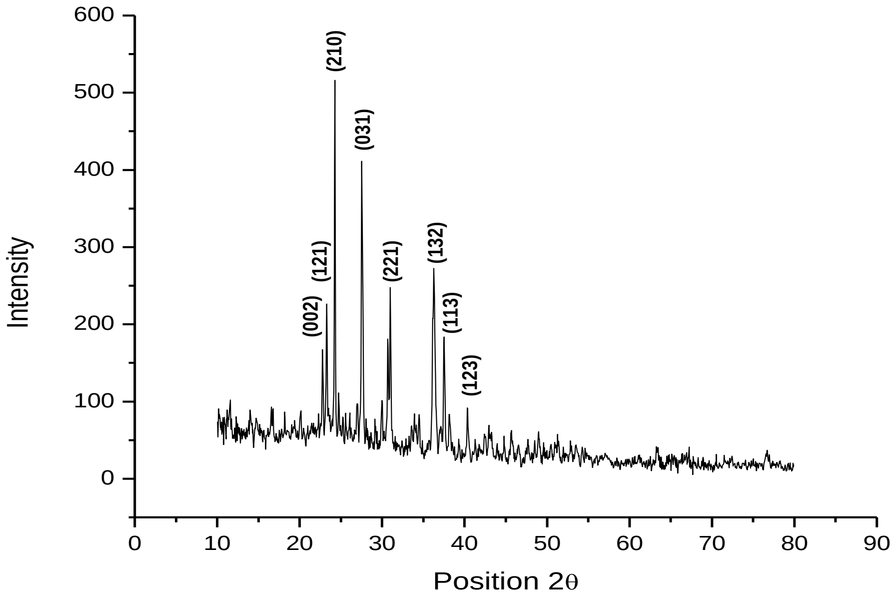

Fig-9: Powder X-ray diffractograms of Zinc Potassium Hydrogen tartrate

From the diffractograms, it is clear that the entry of Zn^{2+} ions in the modified composition of potassium hydrogen tartrate crystals lead to shift in the positions of peaks which shows that the doping has brought about a change in the internal structure of crystals due to change in bond lengths [15].

The indexed XRD data for Zinc doped potassium hydrogen tartrate crystal is presented in Table 3.

The calculation of cell parameters reveals that Zinc doped crystals belong to orthorhombic crystal system having space group space $P2_12_12_1$.

A comparison of crystallographic data of pure and doped crystals are given in Table 4.

Table-3: The indexed XRD data for Zinc doped potassium hydrogen tartrate crystal

2θ value of Pure $KHC_4H_40_6$	2θ value of Zinc doped $KHC_4H_40_6$
23.3000	23.259
23.7512	24.245
24.7170	24.730
28.0300	27.538
31.3039	30.971
36.7210	36.241
37.9800	37.488

Table-4: A comparison of crystallographic data of pure and doped crystals

CHEMICAL FORMULA	**INTER AXIAL ANGLES**	**UNIT CELL DIMENSIONS (Å)**	**UNIT CELL VOLUME ($Å^3$)**
Pure $KHC_4H_40_6$	$\alpha=\beta=\gamma=90^0$	a=9.625 b=8.545 c=10.425	857.41
Zinc doped $KHC_4H_40_6$	$\alpha=\beta=\gamma=90^0$	a=7.61794 b=10.58691 c=7.81598	630.31

It is clear that doping has brought about a change in the cell dimensions [16].

The cell parameters of pure crystal are worked out to be a=9.625 Å, b=8.545 Å, c=10.425 Å, $\alpha=\beta=\gamma=90^0$ while that of zinc doped potassium hydrogen tartrate crystal a=7.61794 Å, b=10.58691 Å, c=7.81598 Å, $\alpha=\beta=\gamma=90^0$ [16, 17].

ANTIMICROBIAL ACTIVITY

Antibacterial activity of different chemical compound preparation against pathogenic Bacterial strains were obtained from the Department of Biotechnology, Bishop Heber College.

The antimicrobial activity of zinc doped potassium hydrogen tartrate against five different bacteria namely Klebsiella, Pseudomonas, E.coli, Staphylococcus and Proteus by well diffusion method. That five different bacteria has been tested as per the zone of inhibition formation. There were considerable variation in the antibacterial potency of the sample.

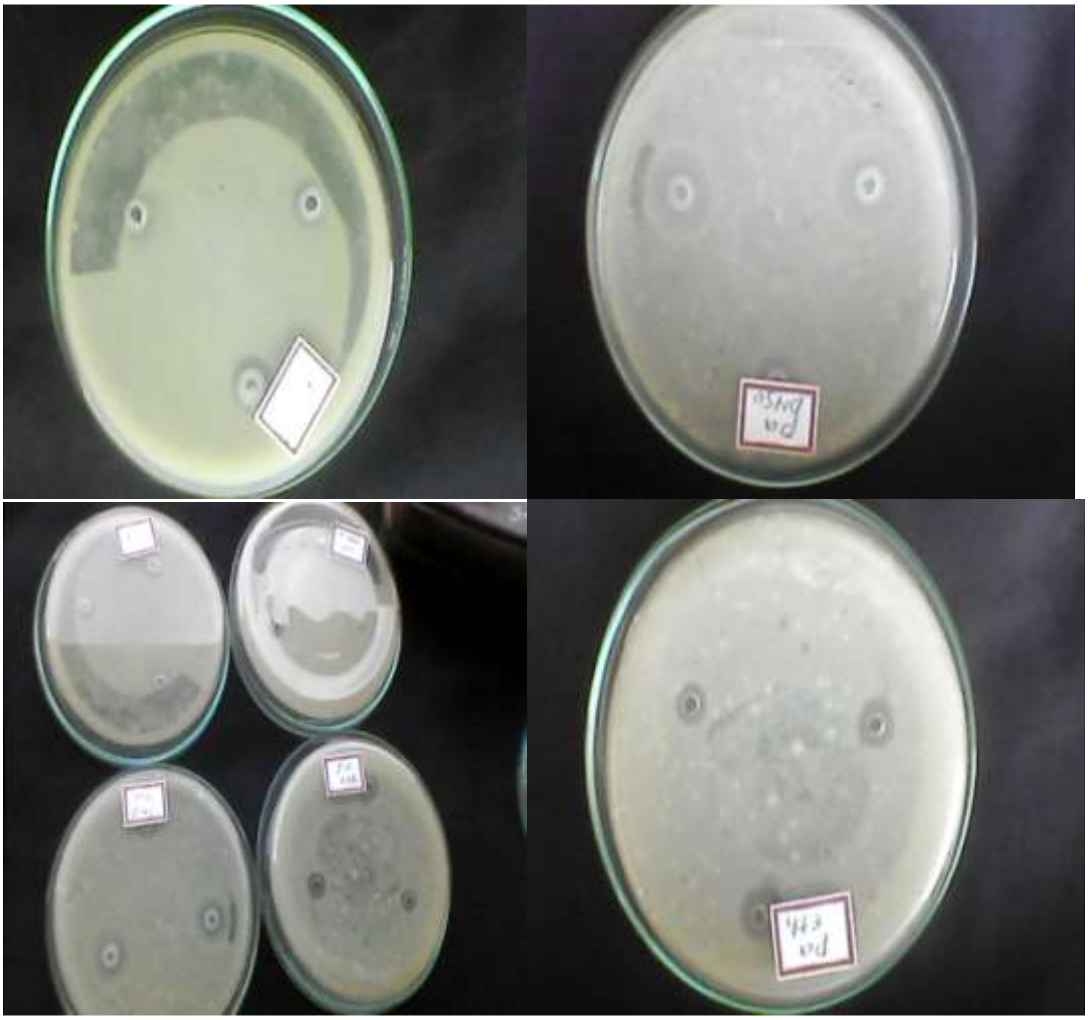

Fig-10: Well Diffusion Method of zinc doped $KHC_4H_40_6$ sample

Klebsiella (0.8mm), Pseudomonas (0.7mm) and E.coli (0.6mm) were inhibited in the descending order of potency of zinc doped potassium hydrogen tartrate was nontoxic to proteus and staphylococcus.

Table 5 Antibacterial activity of different chemical compound preparation against pathogenic Bacterial strains

S. No	Name of the bacteria	Mean zone of inhibition in mm Zinc doped potassium hydrogen tartrate
01	Klebsiella	0.8 mm
02	Pseudomonas	0.7 mm
03	E.coli	0.6 mm
04	Staphylococcus	-
05	Proteus	-

The results clearly indicate that the antibacterial activities against the tested bacteria. Zinc doped potassium tartrate crystal toxic for Klebsiella, pseudomonas and E.coli bacteria. There are in human body and create some disease like Bloody diarrhea, Stomachcramps and Vomiting. The crystal is convert to medical application to give human body to control that bacteria.

CONCLUSION

The Powder XRD patterns of the grown crystal were obtained using PANalytical XPERT-PRO diffractometer with Cu kα radiation (λ=1.5406 Å). The C and H contents in the crystals were determined and identification of metallic elements in the zinc doped crystal were detected by using an energy dispersive spectrometer Fei quanta 250 system.

The FTIR spectra of the material under study in the range of (4000-500 cm^{-1}) were recorded on the Thermo Nicolet spectrometer. Raman spectra were measured using Cray spectometer. The high efficiency of the PL suggests a good potential of this crystal as well as for photonics application. Antibacterial test organisms used in the study were E.coli, Proteus, Staphylococcus, Klebsiella and Pseudomonas. The entire test cultures were obtained from the Biotechnology Laboratory of Bishop Heber College, authenticated cultures were checked for purity before use.

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